भारतीय मानक
Indian Standard

IS 17441 (Part 2) : 2021

निस्तापित एल्युमिना — विशिष्टि

भाग 2 धातुकर्म ग्रेड

Calcined Alumina — Specification

Part 2 Metallurgical Grade

ICS 71.060

© BIS 2021

भारतीय मानक ब्यूरो
BUREAU OF INDIAN STANDARDS
मानक भवन, 9 बहादुरशाह ज़फर मार्ग, नई दिल्ली – 110002
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI-110002
www.bis.gov.in www.standardsbis.in

December 2021 **Price Group 7**

Inorganic Chemicals Sectional Committee, CHD 01

FOREWORD

This Indian Standard (Part 2) was adopted by the Bureau of Indian Standards, after the draft finalized by the Inorganic Chemicals Sectional Committee had been approved by the Chemical Division Council.

Keeping in view the requirements of the Industry and based on the inputs received from Aluminium Industry, the concerned Sectional Committee decided to segregate the Standard on Calcined Alumina into 2 parts:

Part 1 Non-metallurgical grade calcined alumina

Part 2 Metallurgical grade calcined alumina

This standard covers the requirements and method of test for metallurgical grade calcined alumina. Metallurgical grade calcined alumina is aluminium oxide that has been produced by heating aluminium hydroxide, commonly known as hydrate ($Al_2O_3.3H_2O$), at temperature in excess of 1050°C to drive off nearly all chemically combined water. In aluminium hydroxide form, alumina has higher chemically combined water. In calcined form, alumina has higher chemical purity, extreme hardness, high density and a high melting point (slightly above 2050 °C). The metallurgical grade alumina contains predominantly gamma – phase and is used for the separation of Aluminium by smelting. However, by controlling the calcinations temperature and time, properties of alumina can be controlled. With increasing the temperature during calcinations, alumina transforms to α-alumina which is chemically stable with high melting temperature.

Metallurgical grade calcined alumina are manufactured using the stationary flash calciner, gas suspension, fluid bed or rotary. The residence time in the flash calciner is very low that the temperature sensitive gamma phase is maintained.

In the preparation of this Standard, considerable assistance has been derived from ISO 23201.

The requirements and methods of test for non-metallurgical grade calcined alumina are given in IS 17441 (Part 1).

The composition of committee responsible for the formulation of this standard is given at Annex B.

For the purpose of deciding whether a particular requirement of this standard is complied with the final value, observed or calculated, expressing the result of a test or analysis shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

CALCINED ALUMINA — SPECIFICATION

PART 2 METALLURGICAL GRADE

1 SCOPE

This standard (Part 2) covers the requirements and method of test for metallurgical grade calcined alumina.

2 REFERENCES

The Indian Standards given below contain provisions which through reference in this text, constitute provision of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards.

IS No.	*Title*
264 : 2005	Nitric acid — Specification (*third revision*)
266 : 1993	Sulphuric acid — Specification
1070 : 1992	Reagent grade water (*third revision*)
4200 : 2020	Sodium aluminate (*second revision*)

3 GRADING

There is only one grade of metallurgical calcined alumina. The grade name can be provided by the manufacturer.

4 REQUIREMENTS

4.1 The material shall consist essentially of metallurgical calcined alumina which shall be in the form of a white powder. Material shall also comply with the requirements given in Table 1.

4.2 The material shall comply with all the requirements given in Table 1 when tested in accordance with the test methods given in Annex A.

4.3 Physical Parameters

These parameters depend upon the process involved and controls during production and should be optional (*see* Table 2).

5 PACKING AND MARKING

5.1 Packing

The material shall be packed in clean, dry bulk bags or as agreed to between the purchaser and the supplier. The bags shall not be opened until required for use and shall not remain open for a period longer than required for taking out the material.

5.2 Marking

5.2.1 Each bag shall bear legibly and indelibly the following information:

a) Name and grade of the material;
b) Indication of the source of manufacture; and
c) Mass of the material in the bulk bag

5.2.2 *BIS Certification Marking*

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act*, 2016 and the Rules and Regulations framed thereunder, and the products may be marked with the Standard Mark.

Table 1 Requirements for Metallurgical Grade Calcined Alumina

(*Clause* 4.1)

Sl No.	**Characteristic**	**Requirements**	**Method of Test, Reference to Clause of Annex A**
(1)	(2)	(3)	(4)
(i)	Aluminium oxide, Al_2O_3 (percent by mass, *Min*) dried basis	98.0	A-2 and A-11
(ii)	Sodium oxide (Na_2O), (percent by mass), percent	0.2-0.6	A-3 and A-11
(iii)	Silica (SiO_2), (percent by mass, *Max*), percent	0.04	A-4
(iv)	Iron oxide (Fe_2O_3), (percent by mass, *Max*), percent	0.04	A-5 and A-11
(v)	Calcium (CaO), (percent by mass, *Max*), percent	0.07	A-6 and A-11
(vii)	Loss on ignition (percent by mass, 300-1000 °C, *Max*), percent	1.2	A-7
(vii)	Alpha content, percent	As agreed between the interested parties (typical values < 12)	A-12

Table 2 Requirements for Physical Parameters

(*Clause* 4.3)

Sl No.	**Characteristic**	**Requirements**	**Method of Test, Reference to Clause of Annex A**
(1)	(2)	(3)	(4)
i)	Bulk density (g/cm^3), *Min*	Agreed between interested parties (typical value 0.9)	A-8
ii)	SSA, m^2/g	Agreed between interested parties (typical values 60-100)	A-9
iii)	Screen size, -325 mesh, percent	Agreed between interested parties (typical values < 15 percent)	A-10

ANNEX A

(*Clause* 4.2, *Table* 1)

A-1 QUALITY OF REAGENTS

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be used in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2 DETERMINATION OF ALUMINIUM OXIDE

A-2.1 Outline of the Method

From an aliquot of the main solution, after dilution iron and titanium are precipitated by treatment with caustic soda solution. The precipitate is filtered through dry filter and alumina in the filtrate is determined complexometrically.

A-2.2 Reagents

A-2.2.1 *Caustic Soda* — 20 percent (*m*/*v*).

A-2.2.2 *Concentrated Hydrochloric Acid*-rd = 1.16

A-2.2.3 *Sodium Carbonate* — Solid.

A-2.2.4 *EDTA* (0.05 M) — Dissolve about 18.64 g of the disodium salt of EDTA in water and transfer to a 1000 ml volumetric flask, dilute to the mark and mix.

A-2.2.5 *Methyl Red Indicator Solution* — 0.1 percent (*m*/*v*) in rectified spirit.

A-2.2.6 *Dilute Hydrochloric Acid* — 1 : 1 (*v*/*v*).

A-2.2.7 *Buffer Solution* (*p*H 5.3) — Dissolve 21.5 g of sodium acetate ($CH_3COONa.3H_2O$) in 300 ml water, add 2 ml glacial acetic acid and dilute to one litre.

A-2.2.8 *Standard Zinc Solution* (0.05 M) — Dissolve 3.26 g of electrolytic zinc granules (99.95 percent purity) in dilute hydrochloric acid (1 : 1) and evaporate the soultion to 1 to 2 ml, dilute with water add 50 ml buffer solution (*p*H 5.3), mix and make up the volume to one litre. Standardize against standard aluminium solution following the method for determination of aluminium.

A-2.2.9 *Standard Aluminium Solution* (0.05 M) — Weigh accurately 1.3490 g of pure aluminium wire (99.9 percent) in a 500 ml beaker containing 50 ml of water and 50 ml concentrated hydrochloric acid. Heat the solution to dissolve the metal. Cool and dilute the solution to 1 000 ml in a volumetric flask (1 ml = 0.002 550 g $A1_20_3$).

A-2.2.10 *Xylenol Orange Solution* — 0.5 percent (*m*/*v*) in water acidified with 2 drops of dilute hydrochloric acid (40 percent).

A-2.2.11 *Ammonium Fluoride* — Solid.

A-2.3 Procedure

A-2.3.1 Pipette out 50 ml aliquot from the main solution into 400 ml beaker and neutralize the solution with caustic soda solution. Dissolve the precipitate formed in minimum quantity of concentrated hydrochloric acid and pour the solution slowly with constant stirring to a 200 ml volumetric flask containing 25 ml of caustic soda solution and 1 g sodium carbonate. Keep this on the water-bath for about an hour and then cool to room temperature. Make up the volume to the mark with water and shake thoroughly. Filter dry and collect the filtrate in a dry beaker or dry polythene bottle.

A-2.3.2 Pipette out an aliquot (100 ml) from the filtrate to a 250 ml conical flask. Add about 30 ml of 0.05 M EDTA solution and a drop of methyl red indicator. Neutralize the solution with dilute hydrochloric acid, add 15 ml buffer solution and boil the solution for about 2 min. Cool to room temperature and titrate with standard zinc solution using 2 to 3 drops of xylenol orange indicator. The colour changes from yellow to red. Add about 2 g of ammonium fluoride to the solution and boil the solution for about 2 min. Add 10 ml buffer solution cool to the room temperature and then titrate with standard zinc solution adding a drop of xylenol orange indicator. The end point is from yellow to pinkish red. The percentage of alumina is calculated from the titre value of second titration with zinc.

A-2.4 Calculation

$$\text{Alumina } (Al_2O_3)\text{, percent} = \frac{A \times B}{C} \times 100$$

A = ml of zinc solution required for second titration,

B = equivalent Al_2O_3 (in g) per ml of zinc solution, and

C = mass in g of the sample as represented by the aliquot taken.

NOTE — This method is applicable when CaO and MgO is < 0.3 percent (m/m).

A-2.5 Alternatively, aluminium oxide can be determined using instrumental test method given at **A-11**.

A-3 DETERMINATION OF SODIUM OXIDE (NA_2O)

A-3.1 Apparatus

A-3.1.1 *Flame Photometer,* along with digital reading output.

A-3.2 Reagents

A-3.2.1 *Concentrated Nitric Acid* (*see* IS 264).

A-3.3 Procedure

Weigh 0.5 g of the sample into a 250 ml platinum dish or crucible. Add 10 ml of water and 10 ml of concentrated nitric acid and cover the dish. Evaporate the contents to dryness on a hot plate. Add another 10 ml of concentrated nitric acid and evaporate to dryness again. Extract the residue with 20 ml of nitric acid (1 : 1) by gentle boiling followed by washing the cover with minimum amount of acid solution and take the wash into the dish.

Filter through Whatman No. 40 or equivalent filter paper into a 250 ml volumetric flask. Wash three times with hot water. Cool the flask and dilute to the mark with water. Take 25 ml of the solution and dilute to 100 ml in a volumetric flask.

Aspirate the solution to flame photometer and read the intensity of sodium emission. The instrument shall be calibrated in the range of 0 to 5 mg/l of sodium and the calibration curve shall be plotted with concentration of x-axis and emission intensity on y-axis.

A-3.4 Calculation

$$\text{Sodium and its compounds (as } Na_2O\text{), percent by mass} = X \times 0.2$$

where

X = mg/l of Na_2O from the calibration curve.

A-3.5 Alternatively, Sodium oxide can be determined using instrumental test method given at **A-11**.

A-4 DETERMINATION OF SILICA (as SiO_2)

A-4.1 Principle

The sample is decomposed with an acid mixture and evaporated to fumes. The fumed mass is extracted with water, boiled to dissolve the salts and filtered. The residue is ignited and silica is determined through hydrofluorization.

A-4.2 Reagents

A-4.2.1 *Concentrated Sulphuric Acid*, rd = 1.84 (*see* IS 266).

A-4.2.2 *Concentrated Nitric Acid*, rd= 1.42.

A-4.2.3 *Concentrated Hydrochloric Acid*, rd= 1.16.

A-4.2.4 *Dilute Sulphuric Acid* = 1 : 20 (*v*/*v*) and 1 : 4 (*v*/*v*).

A-4.2.5 *Sulphuric Acid Washing Solution* = 1 : 25 (*v*/*v*).

A-4.2.6 *Sodium Carbonate (Anhydrous)*

A-4.2.7 *Hydrofluoric Acid*, 40 percent.

A-4.2.8 *Mixed Acids* — Add slowly 150 ml of concentrated sulphuric acid to 450 ml of water while stirring cool and add 200 ml of concentrated hydrochloric acid and 200 ml of concentrated nitric acid.

A-4.3 Procedure

A-4.3.1 Transfer 1.00 g of accurately weighed sample to a 400 ml pyrex beaker. Add 100 ml of freshly prepared mixed acids, cover at low heat till the brown fumes have disappeared. Remove the cover and wash the sides of beaker with little water. Continue heating until the fumes of sulphur trioxide (SO_3) are given off. After cooling to room temperature add about 100 ml of dilute sulphuric acid (1 : 20) and bring to boiling temperature on a hot plate and continue boiling till the sulphates goes in the solution leaving silica and if any unattacked sample. Allow to settle, filter (use Whatman filter No. 40 with pulp) and wash the residue and the filter paper with hot washing solution until free from dissolved salts, collect the filtrated and washings in 500 ml beaker.

A-4.3.2 Transfer the filter paper in a platinum crucible, ignite in a furnace, cool the ignited residue (containing a little of undissolved sample and silica) and mix with 2 g of sodium carbonate. Cover the platinum crucible with lid, heat slowly on low gas flame in the beginning and increase the temperature with intermittent swirling of the sample. Finally put the lid and keep in a furnace at 1 000 °C for about 15 minutes. Remove and cool the crucible to room temperature and dip the crucible along with lid in the beaker containing main solution A. The cake is disintegrated with glass rod and removed from the crucible. Wash the crucible and lid and collect the washings in the sample Solution A. The main solution is evaporated slowly and heating is continued till the fumes of sulphur trioxide (SO_3) are given off. Cool and add 100 ml of dilute sulphuric acid (1 : 20) from the sides of beaker and bring to boiling on a hot plate to dissolve all the solids other than dehydrated silica. Allow to settle, filter (use Whatman filter paper No. 40 with pulp) and wash the residue and the filter paper with hot washing solution until free from dissolved salts. Collect the filtrate and washings in 500-ml volumetric flask (Solution A). Transfer the filter paper to a platinum crucible, ignite in furnace to constant mass at 950 to 1 000 °C, cool in a desiccator and weigh. Moisten the precipitate with a few drops of water, add 1 to 2 ml of dilute sulphuric acid (1 : 4) and 10 ml of hydrofluoric acid, evaporate slowly to dryness at a low heat. Repeat the process with the addition of 2 to 5 ml of hydrofluoric acid and evaporate slowly to dryness. Ignite in a furnace at 950 to 1 000 °C for 5 to 10 min, cool in a desiccator and weigh. Preserve the residue.

A-4.3.3 Carry out a blank determination following the same procedure and using the same quantity of all reagents without the sample.

A-4.3.4 *Preparation of Main Solution*

Fuse the residue preserved under **4.3.2** thoroughly with about 1 g of sodium carbonate extract with 20 to 30 ml of dilute sulphuric acid (1 : 20). Add the extract to the filtrate and washings (Solution A) obtained under **4.3.1** and make up the volume up to the mark with water and mix thoroughly.

A-4.4 Calculation

$$\text{Silica, Percent} = \frac{(A - B) - C \times 100}{D}$$

A = mass in g, of platinum crucible with the residue before volatilization;

B = mass in g, of platinum crucible with the residue obtained after repeated volatilization with HF;

C = mass in g, of silica obtained from the blank determination; and

D = mass in g, of the sample taken.

A-5 DETERMINATION OF IRON OXIDE

A-5.1 Principle

Iron oxide is determined spectrophotometrically using o-phenanthroline.

A-5.2 Reagents

A-5.2.1 *Standard Iron Solution (10 µg/ml)* — Dissolve 0.4911 g of ferrous ammonium sulphate [$(FeSO_4).(NH_4)_2SO_4.6H_2O$)] in water. Add 10 ml of 1 : 1 sulphuric acid and dilute with water to 1 000 ml. Pipette out 50 ml of the solution and dilute with water to 500 ml. One millilitre of this solution is equivalent to 0.01 mg of iron.

A-5.2.2 *Sulphuric Acid* — 1 : 1.

A-5.2.3 *Hydroxylamine Hydrochloride Solution* — 10 percent (*m*/*v*),

A-5.2.4 *o-Phenanthroline Solution* — Dissolve 0.1 g of o-phenanthroline in 2 ml of methanol and dilute to 100 ml with water and store in a dark bottle with a ground-glass stopper.

A-5.2.5 *Sodium Acetate Solution — 2 M.*

A-5.3 Procedure

Take 10, 20, 30, 40 and 50 ml aliquots of standard iron solution in separate 100 ml beakers and add reagent grade water to adjust volume approximately to 50 ml, wherever required. Adjust the *p*H between 2 to 3 using 1 : 1 sulphuric acid and then between 3 to 4 using sodium acetate solution. Add 2 ml of hydroxylamine hydrochloride solution and 5 ml of o-phenanthroline solution. Dilute with water and make up the volume in each beaker to 100 ml. Determine the absorbance after 30 min against reagent blank consisting of same amount of all reagent in reagent grade water at 510 nm using a spectrophotometer. Plot a calibration curve of absorbance against concentration of 1 µg/ml to 5 µg/ml as obtained by following the above dilution steps using the standard iron solution.

Transfer 1.00 g of accurately weighed sample to a 400 ml pyrex beaker. Add 100 ml of freshly prepared mixed acids, cover it and give low heat till the brown fumes have disappeared. Remove the cover and wash the sides of beaker with little water. Continue heating until the fumes of sulphur trioxide (SO_3) are given off. After cooling to room temperature add about 100 ml of dilute sulphuric acid (1 : 20) and bring to boiling temperature on a hot plate and continue boiling till the sulphates goes in the solution leaving silica and if any unattacked sample. Allow to settle, filter (use Whatman filter No. 40 with pulp) and wash the residue and the filter paper with hot washing solution until free from dissolved salts, collect the filtrated and washings in 500 ml beaker.

Take 10 ml aliquot in a 100 ml beaker. Adjust the *p*H to 2 to 3 using 1 : 1 sulphuric acid and then between 3 to 4 using sodium acetate solution. Add 2 ml of hydroxylamine hydrochloride solution and 5 ml of o-phenanthroline solution. Dilute with water the contents of the beaker to 100 ml. Determine the absorbance after 30 min against reagent blank at 510 nm using a spectrophotometer. Read the concentration for the absorbance determined using the calibration curve.

A-5.4 Calculation

Iron oxide (as Fe_20_3), percent by mass = µg/ml of Fe_2O_3 from the calibration curve × 0.05

A-5.5 Alternatively, iron oxide can be determined using instrumental test method given at **A-11**.

A-6 DETERMINATION OF CALCIUM (AS CAO)

A-6.1 Outline of the Method

Sample is fused with sodium carbonate and boric acid and dissolved in hydrochloric acid and hydrogen-peroxide. Suitable aliquots are aspirated into flame of atomic absorption spectrophotometer using nitrous oxide-acetylene burner for the determination of calcium.

A-6.2 Reagents

Sodium carbonate, anhydrous, boric acid, solid, concentrated hydrochloric acid (rd = 1.16), hydrogen peroxide, 30 percent (*v*/*v*).

A-6.2.1 *Background Solution*

Weigh 5.72 g of super purity-aluminium turnings. Dissolve in 200 ml of NaOH solution (5 N). When the aluminium has dissolved, add 20.0 g of boric acid and 400 ml of concentrated hydrochloric acid. Transfer to a 1 000 ml volumetric flask, dilute to volume with water and mix. Store in a polyethylene bottle. Dissolve 2.50 g of iron powder in 100 ml dilute hydrochloric acid (1 : 1). Warm to complete dissolution. Transfer to a 250 ml volumetric flask, dilute with water and mix. This solution contains 10 mg Fe/ml.

Dissolve 2.50 g of iron powder in 100 ml dilute hydrochloric acid (1 : 1). Warm to complete dissolution. Transfer to a 250 ml volumetric flask, dilute with water and mix. This solution contains 10 mg Fe/ml.

Dissolve 7.4 g of potassium titanium oxalate in 300 ml of water. Transfer to a 500 ml volumetric flask, dilute to volume with water and mix. This solution contains 2 mg Ti/ml.

In a stainless steel beaker, dissolve 1.392 g of silicic acid in 50 ml of NaOH solution (5 N). Warm if necessary to complete dissolution. Transfer to 500 ml polyethylene volumetric flask, dilute to volume with water and mix. Store in a polyethylene bottle. This solution contains 1 mg Si/ml.

Dissolve 26.6 g of lanthanum chloride heptahydrate ($LaCl_2.7H_2O$) in water, dilute to 100 ml and mix. This solution contains 100 mg of La/ml.

Into each of the 250 ml volumetric flasks set aside, for the preparation of the calibration solutions, pipette the following:

a) 50 ml of aluminium-boric acid solution;
b) 10 ml of iron solution;
c) 10 ml of titania solution;
d) 10 ml of silica solution; and
e) 50 ml of lanthanum solution.

A-6.2.2 *Standard Calcium Solution, (100 and 10 mg of Ca/litre)*

Weigh 0.624 g of pure calcium carbonate in a 250 ml beaker, add 25 ml of water and 10 ml of concentrated hydrochloric acid. When the salt has dissolved, transfer in 250 ml volumetric flask, dilute with water and mix. Store in a polyethylene bottle. This solution contains 1000 mg Ca per litre.

Transfer 25 ml of this solution in a 250 ml volumetric volumetric flask. Transfer into a polyethylene bottle. This solution contains 100 mg Ca per litre (B).

Transfer 25 ml of solution (B) to 250 ml volumetric flask. Dilute with water and mix. Transfer it to a polyethylene bottle. This solution contains 10 mg Ca per litre.

A-6.3 Procedure

A-6.3.1 Weigh 1 g of sample in a platinum crucible. Add 3.5 g of sodium carbonate-boric acid fusion mixture (2 : 1) and mix thoroughly by stirring with platinum rod. Place the crucible in a muffle furnace at 800 °C and immediately raise the temperature to 1000 °C. Keep the crucible in the furnace for 1 h. Cool the crucible.

A-6.3.2 Strike the bottom of the cruciblc with the wooden handle of a spatula to crack the melt. Dislodge the solids by hitting the sides of the crucible with the wooden handle of the spatula and by gently pushing in the bottom and the sides. Fill the crucible with water and gently heat on a hot plate until the melt has softened. Transfer the mass to a 250 ml beaker using a gentle hot water jet. Add to the crucible a few ml of hot water, 5 ml of concentrated hydrochloric acid and 2 ml of hydrogen peroxide.

Gently heat on a hot plate until the residue is well dissolved. Remove the last traces of melt adhering to the sides of the crucible by adding 10 ml of water and 15 ml of concentrated hydrochloric acid. Heat the crucible on the edge of a hot plate until the solids have dissolved. Transfer the contents of the crucible to the 250 ml beaker. Add 2 ml of hydrogen peroxide.

Heat the contents of the beaker gently on the edge of the hot plate until the evolution of small bubbles has stopped.

NOTES

1 Do not heat strongly (silica will be precipitated). Continue heating if necessary until all the melt is dissolved. Transfer the contents of the beaker to a 250 ml volumetric flask, dilute with water and mix. Transfer 25 ml of the solution into a 50 ml volumetric flask. Dilute to volume with water and mix. (Final test solution).

2 For ores having higher calcium contents than 0.84 percent CaO, an intermediate dilution step prior to the 25ml/50 ml of final dilution is required (for example 1/5 or 1/2.5)

A-6.3.3 *Blank*

Prepare the reagent blank using 3.5 g of fusion mixture and following the same procedure as specified above.

A-6.3.4 *Calibration Solution*

To 130 ml (50 + 10 +10 + 10 + 50 ml) of background solution add the volume of respective standard solution given in Table 3. Dilute to 250 ml in volumetric flasks.

Transfer 25 ml of each of the solutions to dry 50 ml volumetric flasks. Dilute to volume with water and mix. These solutions are the final calibration solutions.

NOTE — The range of calcium that can be covered may vary from instrument to instrument. More diluted or more concentrated calibration solutions can be used by adjusting accordingly the portions of standard solutions required.

Table 3 Standard Solutions (Calcium) for Calibration Curve

(*Clause* A-6.3.4)

Percent CaO	Volume in ml of Standard Solution 10 mg Ca/L	Volume in ml of Standard Solution 100 mg Ca/L
0.014	10.0	0
0.028	20.0	0
0.070	0	5.0
0.140	0	10.0
0.210	0	15.0
0.280	0	20.0
0.560	0	40.0
0.840	0	60.0

A-6.3.5 *Zero Calibration Solution*

Transfer 130 ml (50 + 10 + 10 + 10 + 50 ml) of background solution into 250 ml volumetric flasks, dilute to volume and mix. Transfer 25 ml of this solution into 50 ml volumetric flask, dilute and mix.

A-6.3.6 *Adjustment of Atomic Absorption Spectrometer*

Fit the burner in accordance with manufacturer's instructions. Light nitrous oxide-acetylene flame. Set the wavelength for magnesium (285.2 nm) and calcium (422.7 nm). After 10 min preheating of the burner, adjust fuel and burner to obtain the maximum absorbance. Aspirate water and calibration solution, to establish that the absorbance reading is not drifting and then set the initial reading for water to zero absorbance.

A-6.3.7 *Atomic Absorption Measurements*

Aspirate the calibration solutions and the final test solutions in order of increasing absorption starting with diluted blank test solution and the zero calibration solution, with the final test solutions being aspirated at the proper point in the series. Aspirate water between each solution and record the readings when stable responses are obtained. Repeat the measurements twice and average the readings.

Obtain the net absorbance of each calibration solution by subtracting the absorbance of the zero calibration solution. Similarly, obtain the net absorbance of the final test solutions by subtracting the absorbance of the diluted blank test solution.

Prepare a calibration curve by plotting the net absorbance values of the calibration solutions against the concentrations of magnesium or calcium, expressed as percent CaO.

Convert the net absorbance values of the final test solutions to percent CaO by means of the calibration curves.

A-6.3.8 *Calculation of Calcium Content*

The calcium content, as a percentage by mass, is calculated from the following formula:

$$\frac{c \times 1.000 \times f}{m}$$

where

c = concentration, expressed as percent CaO, in 1.000 0 g test portion;
m = exact mass, in grams, of test portion; and
f = dilution factor.

If any further dilution is required then use the dilutions described.

A-6.3.8.1 From the calcium content calculated above, the percentage of calcium oxide is calculated as follows:

$$\% \text{ Ca} \times 1.39 = \% \text{ CaO}$$

A-6.4 Alternatively, Calcium can be determined using instrumental test method given at **A-11**.

A-7 LOSS ON IGNITION

A-7.1 Apparatus

A-7.1.1 *Platinum Crucible*

A-7.1.2 *Muffle Furnace*

A-7.2 Procedure

Weigh accurately 5.00 +–0.5 g of sample into a previously weighed platinum crucible. Heat slowly first and then at a gradually increasing temperature. It should be dried at 300 °C for two hours and finally ignite at 1 000 °C for two hours. Cool in a desiccator containing preferably magnesium perchlorate and weigh. Repeat the heating till constant weight.

A-7.3 Calculation

$$\text{Loss on heating, percent by mass} = \frac{(A - B)}{m} \times 100$$

where

A = mass in g, of the crucible with sample at 300 °C;
B = final mass in g, of crucible with residue after ignition at 1 000 °C; and
C = mass in g, of the sample taken.

A-8 BULK DENSITY

A-8.1 Apparatus

A-8.1.1 funnel, of diameter 10 cm and angle 60°, with a stem of length 8 mm and aperture diameter of 6 mm.

A-8.1.2 Cylindrical container, of capacity approximately 200 ml, with a ratio of internal diameter to internal length l/6 approximately.

A-8.1.3 Suitable stand and ring support, to allow the funnel to be set to a predetermined height above the top level of the cylindrical container.

A-8.2 Procedure

Use the crude sample and set the cylindrical container on a flat base. Adjust the funnel so that its axis coincides approximately with that of the cylinder, with the tip of the funnel at a height of 10 cm above the cylindrical container top. Feed the aluminum oxide into the center of the funnel at about 40 mm above the funnel so as not to communicate any vibration to the apparatus, at about 20 to 60 g/min. If blocking occurs at the stem, facilitate the passage of the aluminium oxide with a piece of wire, taking care not to vibrate the cylindrical container. Stop the feed when the aluminium oxide has formed a cone above the top level of the cylindrical container and is spilling over. Remove the cone of surplus aluminium oxide by gently drawing a straight edge across the top rim of the cylindrical container, without communicating any vibration to the latter. Weigh the cylindrical container and its contents.

A-8.3 Calculation

The untamped density is given by the formula =

$$\frac{(m_2 - m_o)}{(m_1 - m_o)}$$

where

m_o = mass, in grams, of the empty cylindrical container;

m_1 = mass, in grams, full of distilled water; and

m_2 = mass, in grams, full of aluminium oxide.

If the cylindrical container used has a capacity of exactly 200 ml, the formula for the calculation of untamped density becomes $(m_2 - m_0)/200$.

It will usually be more convenient to use one particular cylindrical container of capacity approximately 200 ml and determine the value of $(m_1 - m_o)$ to be used in all determinations.

A-9 DETERMINATION OF SURFACE AREA

A-9.1 Apparatus

The apparatus consisting of the following is assembled as shown in Fig. 1.

A-9.2 Procedure

Connect the chamber, flow meters and nitrogen cylinder as shown in Fig. 1. Take the sample in the adsorption cell and regenerate it in the tubular furnace at 250 °C with dry air/nitrogen purge. Cool the sample in an empty desiccator. Adjust the two nitrogen flow inlets to get a partial pressure of 0.05 to 0.075 mm of Hg. Weigh the cooled sample and place it in the adsorption chamber. Carry out the adsorption for two hours till equilibrium is reached. Weigh the cell. Change the partial pressure of benzene to a higher value, say 0.125 to 0.150 and continue the adsorption by replacing the cell in the chamber for 1 hour. Note four readings in the partial pressure range of 0.05 to 0.35 mm of Hg.

A-9.3 Calculation

BET equation is:

$$\frac{P/P_o}{g\left(1-\frac{P}{P_o}\right)} = \frac{1}{gm^C} + \frac{c-1}{gm^C}\,P/P_o$$

where

g = amount of benzene adsorbed at any P/P_o;

g_m = amount of adsorbate (benzene) required to form monolayer on the surface; and

C = constant.

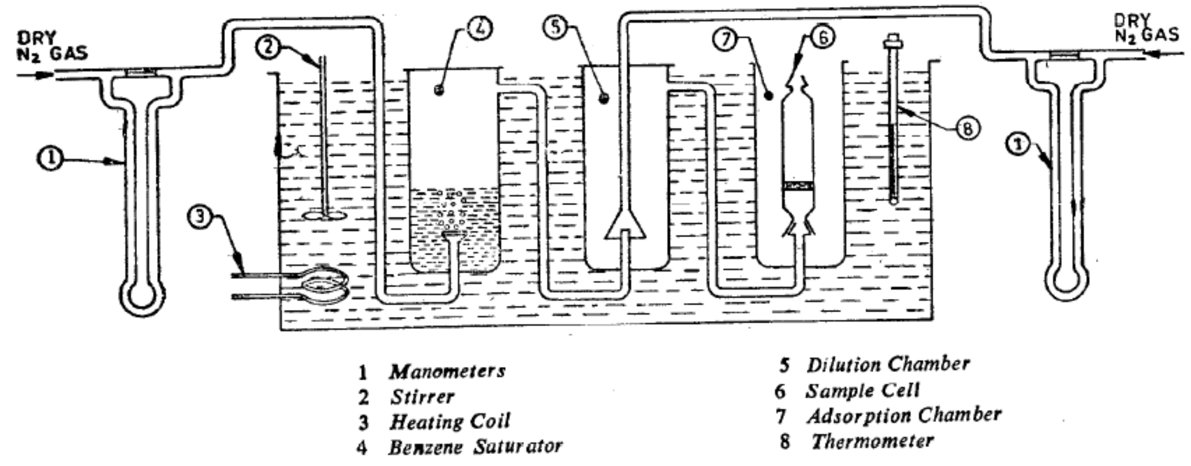

Fig. 1 Apparatus for Surface Area Measurement

If $\frac{P/P_o}{g\left(1-\frac{P}{P_o}\right)}$ is plotted against P/Po, a straight line (BET) plot is obtained in which slope = C-1/g_mC and intercept = 1/g_mC

Slope and Intercept = C-1/g_mC + 1/g_mC – 1/g_m

From the plot, the value of g_m can be obtained.

The constant C = (slope + 1)/Intercept is a measure of the difference in heats of adsorption of the 1st layer and the succeeding layers. The specific surface area is given by:

$$S, m^2/g = g_m.N \times B/m \times M$$

where

N = Avogadros No.;

B = molecular area of the adsorbate (benzene) = 41A^2 = 41 × 10^{-20} m^2;

m = mass of the sample; and

M = molecular mass of benzene.

On substitution, we get, S = g_m × 3166/m

A-9.4 As agreed between the interested parties, the determination of surface area can be done using instrumental method such as surface area analyser by nitrogen adsorption.

A-10 SIEVE ANALYSIS

A-10.1 Normally calcined alumina sieve analysis for 325 mesh (45 micron) will be done on dry basis.

Take 50 gms of dried calcined alumina sample. Select various sieves as per the size fraction required along with 325 mesh sieve. Transfer the 50 gms sample into the sieve set having pan at the bottom.

Using appropriate sieve shaker machine, the sieve set will be vibrated for 20-30 min time at fixed frequency. The vibration time and frequency will be decided based on attrition of material being taken. Weigh each sieve retained material. Convert the retained material weight into percent by dividing each fraction by 50 gms. Report the −325 mesh (−45 micron).

A-10.2 If the sample contains substantial quantity of fine particles, wet method for particle analysis is required. Wetting of particles lowers the surface energy and reduces the force of attraction between the particles. Thus, the particles remain separated. Small amount of surfactants is added in case of liquid with high surface tension.

A-10.2.1 *Procedure*

Take 5 gm of the sample and soak it in suitable solvent. In case of deflocculation, surfactant may be added. Stir the sample and sample is left for soaking period of 10 min. Pass the material through 350 mesh sieve and wash the material until water filtered becomes clear. Collect the sample on 350 mesh sieve and dry it in oven. Collect the retained material on sieve and weigh.

A-10.2.2 *Calculation*

The weight of alumina retained on sieve is calculated as a percentage of the total dry weight of the sample taken.

Percentage retained (percent by mass) = A/M × 100

where

A = mass of residue, in g; and

M = mass of sample, in g.

A-11 ALTERNATE METHOD FOR THE DETERMINATION OF CHEMICAL COMPOSITION OF CALCINED ALUMINA THROUGH X-RAY FLUROSCENCE

A-11.1 Outline of the Method

This method describes the determination of minor and trace elements listed in Table 4 in metallurgical alumina by X-Ray fluorescence on fused samples or pressed sample pallet.

Table 4 Analytical Range

(*Clause* A-11.1)

Parameters	Concentration Range (Percent)
Fe_2O_3	0.0005-0.005
SiO_2	0.003-0.05
TiO_2	0.0005-0.01
CaO	0.0004-0.1
Na_2O	0.1-1.0
P_2O_5	0.0001-0.05
V_2O_5	0.0002-0.01

A-11.2 Apparatus

A-11.2.1 *X-Ray Spectrometer* — X-Ray florescence spectrometer with computing system.

A-11.2.2 *Balance* — Laboratory analytical balance weighing to nearest 0.0001g.

A-11.2.3 *Ball Mill* — Ball Mill with tungsten carbide bowl and balls.

A-11.2.4 *Automatic Fluxer* — Fusion instrument (automatic with 1150 degree C temp).

A-11.2.5 *Drying Oven* — Laboratory drying oven for use up to 150 degree C.

A-11.3 Reagents

A-11.3.1 *Flux* — Anhydrous $LiB_4O_7/LiBO_2$ (lithium tetraborate and lithium metaborate) premixed 12 : 22 proportion or Hoechst wax for powder press.

A-11.3.2 *Lithium Bromide* — LiBR, pure. LiBr solution is prepared by dissolving 30g LiBr in 100 ml of distilled water.

A-11.3.3 *Citric Acid* — Analytical grade. Preparation of 10 percent (w/w) water solution.

A-11.4 Accuracy, Precision, Uncertainty

A-11.4.1 *Accuracy*

Accuracy is expressed as one standard deviation obtained using 16 alumina standards in the regression analysis. The regression procedure calculates the accuracy of the fit (RMS) as follows.

$$\textbf{RMS} = \sqrt{\frac{\sum(\text{C chem} - \text{C calc})}{\text{n} - \text{k}}}$$

where

C chem = known concentration;

C calc = measured concentration;

n = number of standards included; and

k = number of coefficients calculated from regression.

A-11.4.2 *Precision*

t = t10 = 2.228 for 95 percent confidence limit; and

Sr = repeatable standard deviation obtained for an analyte.

Reproducibility represents a combined error of sample homogeneity, preparation and its analysis over a long period of time. The reproducibility R expressed as one standard deviation was obtained for the repetitive analysis (35 times) by 2 operators of ALU-02 standard. The reproducibility R was calculated as follows.

$$\mathbf{R = t\sqrt{2}\ Sr = 2.87\ Sr}$$

where

t = t34 = 2.03 for 95 percent confidence limit, and

Sr = Reproducibility standard deviation.

A-11.4.3 *Uncertainty*

Uncertainty is calculated based on the reproducible data.

A 11.5 Procedure

All samples must be oven dried at 105 ºC for 2 h or longer. Mix the sample properly and obtain analytical portion of the sample material. Weigh approx. 10 g of sample for grinding. Grind the sample material in tungsten carbide bowl with tungsten carbide balls for 10 min. Weigh exactly 2.5 +/– 0.001 g sample portion in the specified platinum crucible. Add exactly 5 +/– 0.001 g of the flux. Add three drops LiBr solution. Fuse the material in automatic fluxer for 25 min at 1100 degree C. Collect the Bead and put the sample no, don’t touch the side exposing to analysis side.

Press pellet preparation:

Grind 8gms sample with 2 gm wax in vibratory cup mill (RPM 1400 ,60 s)

Pellet the sample mix using a pelletizing press (200 KN, 10 s or with required load so that sample is intact).

In XRF spectrometer, arrange the calibration settings for the methods according to manufacturer’s specifications. The X-ray intensity of an element in a sample is dependent on both its concentration and the concentrations of all the other elements making up the sample. Applying matrix corrections compensates for these inter-element effects if required. Calculate the inter-element coefficients (alphas) and the line overlap correction coefficients if required. These coefficients improve the degree of fit in the calibration. Once completed, save the calibration parameters. The instrument will require periodical corrections by use of the monitor samples. Recalibration is not necessary unless concentrations attained for the quality control sample are not within the prescribed tolerance limits. Once fifteen days, run the monitor samples to correct for the instrument drift, then analyze the control sample and the unknown samples.

If the result for the control sample does not agree within the tolerance limit established for each analyte with the concentration obtained during or immediately after calibration, repeat the entire analysis. Calculations are carried out automatically by the instrument’s analytical software. The software contains pre-determined, analytical default conditions for the elements of interest.

Run the prepared alumina samples in the selected alumina programme in the XRF. Once the analysis is over, results will be displayed in the screen. Total Al_2O_3 percentage is calculated with the use of following formula; 100 – total impurities percentage.

A-12 ALPHASE PHASE ANALYSIS THROUGH X-RAY DIFFRACTION

A-12.1 Outline of the Method

This method uses X-Ray diffraction (XRD) to determine alpha alumina in calcined un microfine

alumina samples, in the concentration range of 0 to 100 percent. This method is used for metallurgical grade samples.

A-12.2 Apparatus

A-12.2.1 *X-Ray Diffractometer* — X-Ray diffractometer with computing system.

A-12.2.2 *Balance* — Laboratory analytical balance weighing to nearest 0.0001 g.

A-12.2.3 *Ball Mill* — Ball mill with tungsten carbide bowl and balls.

A-12.2.4 *Calcined Oven Furnace* — Furnace for use upto 650 degree C.

A-12.2.5 *Drying Oven* — Laboratory drying oven for use up to 150 degree C.

A-12.3 Reagents

A-12.3.1 *Ethanol* — Analytical grade

A-12.3.2 *Drift Correction Monitor* — Corundum disk

A-12.4 Accuracy, Precision, Uncertainty

A-12.4.1 *Accuracy*

Accuracy is expressed as one standard deviation obtained using 16 alumina standards in the regression analysis. The regression procedure calculates the accuracy of the fit (RMS) as follows:

$$\textbf{RMS} = \sqrt{\frac{\sum\left(\text{C chem} - \text{C calc}\right)}{\text{n} - \text{k}}}$$

where

C chem = is the known concentration;

C calc = is the measured concentration;

n = is the number of standards included; and

k = is the number of coefficients calculated from regression.

A-12.4.2 *Precision*

Repeatability is expressed as one standard deviation and was calculated from the reputative analysis from the standard:

$$\text{R} = \text{t}\ \sqrt{2}\ \text{Sr}$$

t = t10 = 2.228 for 95 percent confidence limit, and

Sr = repeatable standard deviation obtained for an analyte.

Reproducibility represents a combined error of sample homogeneity, preparation and its analysis over a long period of time. The reproducibility R expressed as one standard deviation was obtained for the repetitive analysis (35 times) by 2 operators of ALU-02 standard. The reproducibility R was calculated as follows.

$$\textbf{R} = \textbf{t}\sqrt{\textbf{2}}\ \textbf{Sr} = \textbf{2.87 Sr}$$

where

t = t34 = 2.03 for 95 percent confidence limit, and

Sr = Reproducibility standard deviation.

A-12.4.3 *Uncertainty*

Uncertainty is calculated based on the reproducible data.

A-12.5 Procedure

A-12.5.1 All samples must be oven dried at 105 ºC for 2 h or longer. Mix the sample properly and obtain analytical portion of the sample material. Weigh approx. 10 g of sample for grinding. Grind the sample material in tungsten carbide bowl with tungsten carbide balls for 4 min. Prepare pallet by using hand press. Run the sample in the calibration program. Using diffractometer software build analytical program as per Table 5.

Table 5 Diffractometer Setting

(*Clause* A-12.5.1)

Radiation	**Co k ,lambda-1.7889 (Bruker)**	**Cu k ,lambda-1.5418 (Panalytical)**
Voltage	40 kv	45 kv
Ampere	35mA	40mA
Div slit	1 mm	1 degree
Scatter slit	2 mm	2 degree
Receiving slit	none	none
Measurement	Fix area	Fix area
Time per step	6 sec	6 sec
Step size	0.02	0.02
Back ground meas	6 sec per peak	6 sec per peak

A-12.5.2 *Data for Intensity Integration*

Assemble standards for application using standard reference material. Prepare linear calibration curves each phase and store the calibration parameter. Run the unknown samples in the prepared calibration method. Evaluate concentration obtained for two reflections.

Reflection	Peak position		Integration range		Background	
	Cu K alpha	Co K alpha	Cu K alpha	Co K alpha	Cu K alpha	Co K alpha
3.48	25.60	29.73	25.2-26.0	29.26-30.22	24.8,26.4	28.8,30.7
1.60	57.50	67.82	56.8-58.2	66.99-68.66	56.4,58.6	66.5,69.14

ANNEX B

(*Foreword*)

COMMITTEE COMPOSITION

Inorganic Chemicals Sectional Committee, CHD 01

Organization	*Representative(s)*
Central Salt and Marine Chemicals Research Institute, Bhavnagar	Dr Kannan Srinivasan (***Chairman***)
Aditya Birla Chemical (I) Ltd, New Delhi	Shri Alok Singh
Alkali Mfrs Association of India, Delhi	Shri K. Srinivasan Shri Subhash Tandon (*Alternate*)
Bhabha Atomic Research Centre, Mumbai	Dr A. V. R. Reddy Dr S. N. Achary (*Alternate*)
Central Drugs Standard Control Organization	Dr Raman Mohan Singh
DGQA, New Delhi	Dr Gurbachan Singh (*Alternate* I) Shri B. S. Tomar (*Alternate* II)
Geological Survey of India, Kolkata	Dr D. K. Das Dr Subhas Chandra (*Alternate*)
Grasim Industries Ltd, Nagda	Shri R. S. Baghel Shri Pankaj Gupta (*Alternate*)
Gujarat Alkalies and Chemicals Ltd, Vadodara	Dr Sunil Sinha
Hindustan Lever Ltd, Mumbai	Vrinda Rajwade Shrimati Poornakala (*Alternate* I) Satyamoorthy (*Alternate* II)
In personal capacity	Dr A. N. Bhat
In personal capacity	Dr T. S. Kathpal
Industrial Carbon Pvt Ltd, Ankleshwar	Shri Rohit Kumar Madhavji Shri Satyan Rohit Kumar (*Alternate*)
Indian Institute of Chemical Technology, Hyderabad	Dr Praveen R. Likhar Dr Rajender Reddy (*Alternate*)
Ministry of Defence (DGQA), Kanpur	Shri R. N. Aparajit
Ministry of Chemicals and Fertilizers	Dr Rohit Misra
National Chemical Laboratory, Pune	Dr Darbha Srinivas Dr Paresh Dhepe (*Alternate*)
National Metallurgical Laboratory, Jamshedpur	Dr Trilochan Mishra Shri Devbrata Mishra (*Alternate*)
National Mineral Development Corporation Ltd, Hyderabad	Shri Rajan Kumar Dr Prashant Sharma (*Alternate*)
National Physical Laboratory, New Delhi	Dr Nahar Singh Dr S. P. Singh (*Alternate*)
National Test House (NR)	Dr Y. C. Nijhawan Dr (Ms) A. Biswas (*Alternate*)
Nirma Chemicals, Ahmedabad	Shri R. A. Joshi Dr K. C. Pathak (*Alternate*)

Organization	*Representative(s)*
Office of the Development Commissioner (MSME), New Delhi	SHRIMATI SHIMLA MEENA SHRI SANTOSH KUMAR (*Alternate*)
Reliance Industries Ltd, Navi Mumbai	SHRI DHANAVADAN MODI
Shriram Institute for Industrial Research, Delhi	DR (MS) LAXMIRAWAT SHRI B. GOVINDAN (*Alternate*)
VOICE, Delhi	SHRI H. WADHWA SHRI K. C. CHAUDHARY (*Alternate*)
In Personal Capacity	SHRI D. K JAIN
Vaibhav Analytical Services, Ahmedabad	SHRI GAURANG OZA
BIS Directorate General	SHRI A K LAL, SCIENTIST 'E' AND HEAD (CHD) [REPRESENTING DIRECTOR GENERAL (*Ex-officio*)]

Member Secretary

SAGAR SINGH
SCIENTIST 'C' (CHD), BIS

Bureau of Indian Standards

BIS is a statutory institution established under the *Bureau of Indian Standards Act*, 2016 to promote harmonious development of the activities of standardization, marking and quality certification of goods and attending to connected matters in the country.

Copyright

BIS has the copyright of all its publications. No part of these publications may be reproduced in any form without the prior permission in writing of BIS. This does not preclude the free use, in the course of implementing the standard, of necessary details, such as symbols and sizes, type or grade designations. Enquiries relating to copyright be addressed to the Director (Publications), BIS.

Review of Indian Standards

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Catalogue' and 'Standards: Monthly Additions'.

This Indian Standard has been developed from Doc No.: CHD 01 (17808).

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110002
Telephones: 2323 0131, 2323 3375, 2323 9402 *Website*: www.bis.gov.in

Regional Offices:		*Telephones*
Central	: Manak Bhavan, 9 Bahadur Shah Zafar Marg NEW DELHI 110002	2323 7617 2323 3841
Eastern	: 1/14 C.I.T. Scheme VII M, V.I.P. Road, Kankurgachi KOLKATA 700054	2337 8499, 2337 8561 2337 8626, 2337 9120
Northern	: Plot No. 4-A, Sector 27-B, Madhya Marg CHANDIGARH 160019	265 0206 265 0290
Southern	: C.I.T. Campus, IV Cross Road, CHENNAI 600113	2254 1216, 2254 1442 2254 2519, 2254 2315
Western	: Manakalaya, E9 MIDC, Marol, Andheri (East) MUMBAI 400093	2832 9295, 2832 7858 2832 7891, 2832 7892

Branches : AHMEDABAD. BENGALURU. BHOPAL. BHUBANESHWAR. COIMBATORE. DEHRADUN. DURGAPUR. FARIDABAD. GHAZIABAD. GUWAHATI. HYDERABAD. JAIPUR. JAMMU. JAMSHEDPUR. KOCHI. LUCKNOW. NAGPUR. PARWANOO. PATNA. PUNE. RAIPUR. RAJKOT. VISAKHAPATNAM.

Published by BIS, New Delhi